

## STATION PROCEDURE COVER SHEET

### A. IDENTIFICATION

Number: CP 807/2807/3807AP

Rev. Q

Title: HYDRAZINE COLORIMETRIC

Prepared By: DAVE PEIFFER

### B. REVIEW

I have reviewed the above procedure and have found it to be satisfactory.

<u>TITLE</u>	<u>SIGNATURE/DATE</u>	<u>TITLE</u>	<u>SIGNATURE/DATE</u>
<u>DEPARTMENT HEAD</u>	<u>[Signature] 8-2-95</u>	_____	_____
<u>Sr. Engineer</u>	<u>[Signature] (8-2-95)</u>	_____	_____

### C. SPECIFIC UNREVIEWED SAFETY QUESTION EVALUATION REQUIRED:

Modifies intent of procedure and changes operation of systems as described in design documents.

YES [ ] NO [X]

(If yes, perform written USQ determination and Safety Evaluation, and contact Manager, Safety Analysis Branch to determine need for Integrated Safety Evaluation.)

#### ENVIRONMENTAL REVIEW REQUIRED

(Adverse environmental impact)

YES [ ] NO [X]

### D. SPECIFIC SAFETY EVALUATION REQUIRED

Affects response of safety systems, performance of systems which may have been credited in the safety analysis or non-credited systems which may indirectly affect safety system response.

YES [ ] NO [X]

(If yes, coordinate Safety Evaluations and contact Manager, Safety Analysis Branch to determine need for Integrated Safety Evaluation.)

### E. INTEGRATED SAFETY EVALUATION REQUIRED

YES [ ] NO [X]

### F. BIENNIAL REVIEW

This revision satisfies biennial review requirements.

YES [X] NO [ ]

### G. PROCEDURE REQUIRES PORC/SORC REVIEW

(In addition to review, items with a YES response must be documented in the PORC/SORC meeting minutes.)

YES [ ] NO [X]

### H. PORC/SORC APPROVAL PORC/SORC Meeting Number Biennial Review (8-2-95)

### I. APPROVAL AND IMPLEMENTATION

The attached procedure is hereby approved, and effective on the date below:

[Signature]  
VICE PRESIDENT/SERVICES/UNIT DIRECTOR

11-17-92  
EFFECTIVE DATE

## STATION PROCEDURE COVER SHEET

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Number: CP 807/2807/3807AP

Rev. 0

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Prepared By: DAVE PEIFFER

### B. REVIEW

I have reviewed the above procedure and have found it to be satisfactory.

<u>TITLE</u>	<u>SIGNATURE/DATE</u>	<u>TITLE</u>	<u>SIGNATURE/DATE</u>
<u>DEPARTMENT HEAD</u>	<u>[Signature] / 10-27-92</u>	<u>Chemist - Unit 2</u>	<u>[Signature] / 10-27-92</u>
_____	_____	_____	_____

### C. SPECIFIC UNREVIEWED SAFETY QUESTION EVALUATION REQUIRED:

Modifies intent of procedure and changes operation of systems as described in design documents.

YES [ ] NO [ ☒ ]

(If yes, perform written USQ determination and Safety Evaluation, and contact Manager, Safety Analysis Branch to determine need for Integrated Safety Evaluation.)

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(Adverse environmental impact)

YES [ ] NO [ ☒ ]

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### E. INTEGRATED SAFETY EVALUATION REQUIRED

YES [ ] NO [ ☒ ]

### F. BIENNIAL REVIEW

This revision satisfies biennial review requirements.

YES [ ☒ ] NO [ ]

### G. PROCEDURE REQUIRES ~~FORC~~ SORC REVIEW

(In addition to review, items with a YES response must be documented in the ~~FORC~~ SORC meeting minutes.)

YES [ ☒ ] NO [ ]

### H. ~~FORC~~ SORC APPROVAL ~~FORC~~ SORC Meeting Number 92-43

### I. APPROVAL AND IMPLEMENTATION

The attached procedure is hereby approved, and effective on the date below:

[Signature]  
VICE PRESIDENT/SERVICES/UNIT DIRECTOR

11/17/92  
EFFECTIVE DATE

## PROCEDURE LEVEL-OF-USE

PROCEDURE TITLE:

Hydrazine Colorimetric

PROCEDURE NUMBER AND REVISION:

CP 807/2807/3807 AP Rev 2

## LEVEL-OF-USE

CONTINUOUS USE

- \* step-by-step use
- \* required in-hand

GENERAL USE

- \* referral prior during performance
- \* required in-field

INFORMATION USE

- \* periodic review
- \* not required in-field
- \* required to perform activities per procedure

## DEPARTMENT HEAD APPROVAL

DEPARTMENT HEAD SIGNATURE:

DATE:

10-27-92

## PROCEDURE USER RESPONSIBILITIES

- PROCEDURE COMPLIANCE - Intent and direction provided in the procedure shall be followed regardless of the level of use. Deviations, except allowed below, are not permitted without an approved change.
- PROCEDURE REVIEW - Steps, notes, cautions, warnings, sequences and expected responses should be understood by personnel prior to starting work, or clarified by appropriate supervision.
- DATA RECORDING SIGN-OFFS - Record data or sign where indicated when the step activity has been completed, unless directed otherwise by the procedure.
- MULTIPLE USERS - Each individual should have a copy to follow if practical. Single master copy available for sign-offs. Sign-offs in master copy completed when activity is completed.
- INADEQUATE UNEXPECTED RESULTS - Stop the task. Place equipment in stable or safe condition. Contact first line supervisor for resolution. Process procedure changes, if needed.
- SEQUENCE OF STEPS - Perform steps in sequence unless otherwise directed within procedure. First line supervisor may waive this requirement if the intent will not be modified or compromised.
- USE OF "N/A" - Use of "N/A" limited to those steps clearly not applicable for a task or when limited portions of procedure are authorized to be completed.
- PARTIAL PERFORMANCE - Sections/porions series-of-steps performance is allowed if approved by first line supervisor.
- JOB EXPERIENCE - Detailed procedure or change is not required if task within a procedure is simple and considered within the skill of the trade. The department head shall determine which tasks may be performed by his personnel without a procedure.
- CONFLICTS - Contact first line supervision to evaluate conflict and initiate changes, if necessary. Changes are made per ACP-QA-3.02.
- ACP CONFLICTS - Resolution of conflict by department head. Submit recommended changes for review and approval. Document resolution and submit to appropriate Director and ACP Procedures group.
- EMERGENCY EVENTS - Emergency or off-normal events not specifically covered in part or total by an approved procedure should be addressed by plant operations personnel.
- ONE-TIME CHANGES - One-time changes are allowed provided the intent is specifically and clearly stated and the change is processed as intent or non-intent per ACP-QA-3.02.

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1. OBJECTIVE

- 1.1 To provide a procedure for the analysis of low hydrazine concentration in the range 5 ppb to 100 ppb.

2. REFERENCES

- 2.1 ASTM Disignation: D1385-67, Hydrazine in Industrial Water.  
2.2 CP 800/2800/3800G Least Square Fit.

3. PREREQUISITES

- 3.1 Power is available to spectrophotometer and the instrument has been properly installed, i.e., isolated from corrosive fumes, mechanical shock and electrical interferences.
- 3.2 Equipment
- Volumetric Flasks 1000, 100 mL
  - Pipets 0.5, 2.5, 5.0, 7.5 10 mL
  - Graduated Cylinder 100, 10 mL
  - Erlenmeyer Flasks<sup>125</sup> 250 mL
  - Cuvettes 4 cm
  - Spectrophotometer

1 chg. 2

4. PRECAUTIONS

- 4.1 ANALYZE the sample as soon after collection as practicable.  
4.2 Sample color and turbidity can interfere with the analysis.

5. PROCEDURE

5.1 PREPARE HCL Solution (1.2 Molar)

- 5.1.1 ADD slowly 100 mL of concentrated HCL to approximately 500 mL demineralized water in a 1000 mL volumetric flask.  
5.1.2 DILUTE to the mark with demineralized water.

5.2 PREPARE Hydrazine Indicator Solution.

- 5.2.1 DISSOLVE 4.0 grams of p-dimethylaminobenzaldehyde in a solution of 200 mL methyl alcohol and 20 mL of concentrated HCL.  
5.2.2 STORE in a dark bottle.  
5.2.3 LABEL with an expiration date two (2) weeks from the preparation date.

### 5.3 PREPARE Standard Hydrazine Solution (1 ppm).

#### NOTE

The 1 ppm solution must be made in two steps:

- 5.3.1 DISSOLVE 0.328 grams of hydrazine dihydrochloride ( $N_2H_4 \cdot 2HCl$ ) in 100 mL of demineralized water and 10 mL of concentrated HCl.
- 5.3.2 DILUTE with demineralized water to 1 liter in a volumetric flask.
- 5.3.3 DILUTE 10 mL of Stock Solution to one liter with demineralized water in a clean volumetric flask.
- 5.3.4 LABEL as a 1 ppm Hydrazine Standard with an expiration date of 3 months from preparation date.
- 5.3.5 DISCARD the remaining 100 ppm stock solution.

### 5.4 PREPARE Calibration Standards

- 5.4.1 DILUTE the following quantities of 1 ppm hydrazine standards and 1.2 mL HCl to 100 mL in volumetric flasks with demineralized water to produce the corresponding concentrations.

<u>mL 1 PPM <math>N_2H_4</math> Std</u>	<u>mL 1:9 HCL Sol</u>	<u>Std Conc. ppb</u>
10.0	5.0	100
7.5	5.0	75
5.0	5.0	50
2.5	5.0	25
0.5	5.0	5

- 5.4.2 LABEL the flasks with an expiration date 1 week from preparation.

### 5.5 PREPARE a Calibration Curve

- 5.5.1 TRANSFER the calibration standards into separate <sup>125 mL or</sup> 250 mL Erlenmeyer flasks. | Ch  
#2
- 5.5.2 ADD 5 mL of HCL solution (1:9) plus 95 mL of demineralized water into a <sup>125 mL or</sup> 250 mL Erlenmeyer flask (zero blank for the spectrophotometer). | Ch  
#2
- 5.5.3 ADD 5 mL of Hydrazine Indicator solution to each flask.
- 5.5.4 MIX thoroughly.
- 5.5.5 WAIT 20 minutes for color development.
- 5.5.6 PLACE the spectrophotometer in the absorbance mode.



- 5.5.7 READ the absorbance of the standards with reference to the water blank at 458 nm.
- 5.5.8 RECORD the absorbance of each standard on Chem. Form 807/2807/3807 AP-1.
- 5.5.9 REPEAT 5.2.1-5.2.6 two additional times.
- 5.5.10 COMPUTE the average absorbance for each standard.

#### NOTE

If directed by Chemistry Supervisor, Use the Least Squares procedure (CP 800/2800/3800 G) to prepare the calibration curve.

If the calibration curve is linear and the absorbance intercept is small, then samples may be measured in the concentration mode with a concentration factor set equal to 1/slope from the linear fit.

A table listing absorbance vs concentration may be used in place of a calibration curve. The concentration values can be calculated by the linear equation  $y = mx + b$  (see procedure 800/2800/3800 G).

- 5.5.11 PLOT the average absorbance vs concentration on linear graph paper.
  - 5.5.11.1 Plot concentration values on the x-axis and absorbance on the y - axis.
  - 5.5.11.2 LABEL calibration curve
    - Title
    - Preparation Date
    - Expiration Date
    - Technician's Initials
- 5.5.12 CHECK the calibration curve with every set of analyses by the use of a control standard.
- 5.5.13 PREPARE a new calibration curve yearly or if there is a significant change in the analytical system.
- 5.6 Sample Collection and Analysis
  - 5.6.1 Sample Collection
    - 5.6.1.1 PLACE 5 mL of HCL solution 1.2 molar in a small bottle.

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- 5.6.1.2 ADD 95 mL of sample to the sample bottle.
- 5.6.1.3 MIX thoroughly
- 5.6.2 Sample Analysis
- 5.6.2.1 PREPARE a control standard and transfer in to a <sup>125 mL or</sup> 250 mL Erlenmeyer flask. | Chg. 2
- 5.6.2.2 PREPARE a spectrophotometer blank as per 5.1.2.
- 5.6.2.3 TRANSFER the collect samples into a separate <sup>250 mL</sup> Erlenmeyer flasks. | Chg. 2  
<sup>125 mL or</sup>
- 5.6.2.4 ADD a 5 mL of Hydrazine Indicator solution to each flask.
- 5.6.2.5 WAIT 20 minutes for color development.
- 5.6.2.6 SET the spectrophotometer to read absorbance or concentration.
- 5.6.2.7 READ the absorbance or concentration of all solutions against the water blank at 458 nm.
- 5.6.2.8 TRANSFER the blank to a clean cuvette.
- 5.6.2.9 TRANSFER each sample to a clean cuvette.
- 5.6.2.10 READ either absorbance or concentration on the spectrophotometer.
- 5.6.2.11 IF operating in the absorbance mode, THEN use the calibration curve to DETERMINED the concentration for all samples.
- 5.6.2.12 RECORD the control standard concentration as read from the calibration curve, on the Control Chart.
- 5.6.2.13 RECORD data on Chem. Form 807/2807/3807-~~3-2~~ | Change #1  
AP-2

## 6. FIGURES

6.1 None

## 7. DISCUSSION

- 7.1 Hydrazine is added to feedwater and closed cooling waters for corrosion control. Analyses are performed for control purposes. A sample is acidified with HCL to prevent hydrazine consumption by oxygen. The reagent para-dimethylamino- benzaldehyde is added to produce a yellow colored complex, where the intensity of the yellow color is proportional to the amount of hydrazine in the water. The color intensity is read on a spectrophotometer at 458 nm wavelength. The upper and lower detection limits are set at 100 ppb and 5 ppb, based upon the high and low calibration points.